# Adhesion, Friction, and Wear of Plasma-Deposited Thin Silicon Nitride Films at Temperatures to 700 °C

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(NASA-IM-101377) ADDESICN, FRICTION, AND N89-11913 WEAR OF FLASMI-IFFOSITED THIN SILICON NITRIDE FILMS AT TEMPERATURES TO 760 C (NASA) 12 p CSCL 11C Unclas G3/27 0174645
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# ADHESION, FRICTION, AND WEAR OF PLASMA-DEPOSITED THIN SILICON NITRIDE FILMS AT TEMPERATURES TO 700 °C

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## **ABSTRACT**

An investigation examined the adhesion, friction, and wear behavior of silicon nitride films deposited by low- and high-frequency plasmas (30 kHz and 13.56 MHz) at various temperatures to 700 °C in vacuum. The results of the investigation indicated that the Si/N ratios were much greater for the films deposited at 13.56 MHz than for those deposited at 30 kHz. Amorphous silicon was present in both low- and high-frequency plasma-deposited silicon nitride films. However, more amorphous silicon occurred in the films deposited at 13.56 MHz than in those deposited at 30 kHz. Temperature significantly influenced adhesion, friction, and wear of the silicon nitride films. Wear occurred in the contact area at high temperature. The wear correlated with the increase in adhesion and friction for the lowand high-frequency plasma-deposited films above 600 and 500 °C, respectively. The low- and high-frequency plasma-deposited thin silicon nitride films exhibited a capability for lubrication (low adhesion and friction) in vacuum at temperatures to 500 and 400 °C, respectively.

## INTRODUCTION

Monolithic silicon nitride and silicon carbide continue to be of great interest for structural use in automobile and aerospace engines because of such properties as high-temperature strength, environmental resistance, and low density. However, the severity of operating conditions in engines has not favored the use of bulk ceramic because of their high sensitivity to microscopic flaws and catastrophic fracture behavior. This brittle nature translates into low reliability for ceramic components and, thus limited application in engines (Kiser et al., 1987; Sliney, 1987; and Brindley, 1987).

For critical automobile and aerospace applications, the components are required, for safety, to display markedly improved toughness and noncatastrophic, or graceful, fracture (Kiser et al., 1987; Sliney, 1987; and Brindley, 1987). Thus, the enhanced interest in

ceramic materials has been further expanded to include fiber-reinforced ceramics and high-performance ceramic-coated materials.

Thin ceramic films are widely used in a variety of applications in which materials in monolithic form are not suitable for diverse and special requirements, and thin films provide the requisite answer. Also, thin ceramic films serve a variety of purposes: providing resistance to abrasion, erosion, corrosion, wear, radiation damage, or high-temperature oxidation; reducing adhesion and friction; and providing lubrication (Buckley, 1981; Czichos, 1984; Hintermann, 1984; Halling and Arnell, 1984; and Schintlmeister et al., 1984).

The present work continues the ongoing tribological studies with thin coating films. Previously, the authors reported the tribiological properties and mechanical strength of thin boron nitride films, which are promising materials for wear-resistant, solid lubricating films (Miyoshi et al., 1985; Miyoshi et al., 1987a; Miyoshi et al., 1987b; and Pouch et al., 1987). The present study concerns thin silicon nitride films. Thin silicon nitride films have been extensively

Thin silicon nitride films have been extensively investigated to fabricate integrated circuits. In open literature, however, their potential as high-temperature solid lubricating films and/or wear-resistant films in tribological systems in both terrestrial and space environments has not been investigated.

The objective of the present study was to investigate the adhesion, friction, and wear behavior of silicon nitride films at various temperatures to 700 °C in vacuum. The films were deposited by low- and high-frequency plasmas (30 kHz and 13.56 MHz).

To minimize coating-substrate interactions and material variables, silicon nitride was plasma-deposited on well-defined pure silicon substrates rather than on engineering alloys such as 440C stainless steel (Valco and Kapoor, 1985: and Valco et al., 1986). The films were analyzed by Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), and ellipsometry. Comparative surface analyses were also conducted with silicon nitride films deposited on gallium arsenide and indium phosphide substrates.

#### **MATERIALS**

Thin films containing silicon nitride were deposited by low- and high-frequency plasmas (30 kHz and 13.56 MHz) on the (100) surfaces of pure silicon, gallium arsenide, and indium phosphide.

Hot-pressed polycrystalline magnesia-doped silicon nitride was used as the pin material in the adhesion and sliding friction experiments.

#### **APPARATUS**

The plasma reactor used to deposit silicon nitride films is described by Valco and Kapoor (1985) and Valco et al., (1986).

The tribological apparatus in this investigation consisted of an ultrahigh vacuum system capable of measuring adhesion and friction at high temperature (Miyoshi et al., 1985; and Miyoshi et al., 1988); the vacuum system contained an XPS spectrometer. The mechanism for measuring adhesion and friction was basically a pin on a flat configuration, as shown schematically in Fig. 1. The flat specimen was mounted on a specimen holder with an electron beam heater assembled in a manipulator. The electron beam heater could raise the temperature of the flat specimen to 1200 °C.

For the adhesion experiments, a manipulator-mounted torsion balance was used (see Fig. 1). The pin specimen was mounted on one end of a movable beam. A free-moving, rod-shaped magnetic core was mounted on the other end of the beam. The coils of a linear variable differential transformer (LVDT) were mounted on a stationary beam. There was no physical contact between the movable magnetic core and the coil structure. The movable beam was supported by a single strand of wire acting as a torsion spring.

For the friction measurements, a manipulator—mounted beam was projected into the vacuum chamber. The beam contained two pairs of flats assembled normal to each other with strain gages mounted thereon. The load was applied by moving the beam normal to the pair of flats that were parallel to each other, and it was sensed by strain gages. The friction force under an applied load was measured during vertical translation by strain gages mounted normal to those measuring the load.

The AES system analyzed the chemical composition of the silicon nitride films (Pouch et al., 1987). The multiple angle, variable wavelength ellipsometer determined refractive indexes, extinction coefficients, and film thicknesses of silicon nitride coatings (Pouch,

et al. 1987).

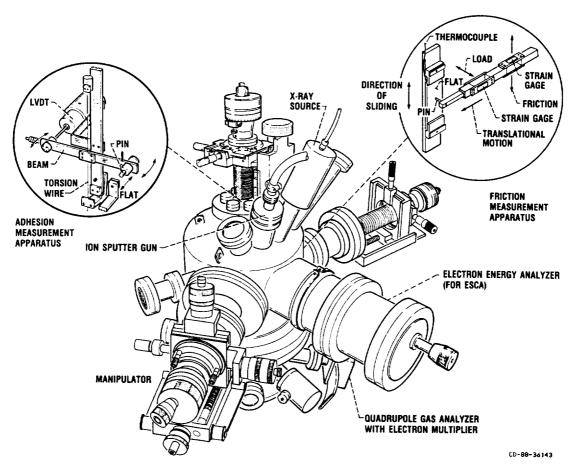


FIG. 1. - APPARATUS FOR MEASURING ADHESION AND FRICTION IN ULTRAHIGH VACUUM.

#### EXPERIMENTAL PROCEDURE

Specimen Preparation

The silicon nitride plasma deposition procedure is described by Valco and Kapoor (1985) and Valco et al. (1986). Silicon nitride coatings were deposited by using a commercial parallel-plate plasma reactor and switching the RF power supply from 30 kHz to 13.56 MHz (Table I). The substrates were placed on the lower, grounded electrode and were heated to a temperature of 300 °C. The flow rates of the silane and ammonia gases (99.9 percent pure) were controlled to maintain the chamber pressure at 500 mtorr. Silicon nitride films of approximately 80 nm were deposited on the 50.8-mm (2-in.) diameter wafers of silicon, gallium arsenide, and indium phosphide, simultaneously.

TABLE I. - PLASMA DEPOSITION OF SILICON NITRIDE FILMS

Substrates			$\overline{\cdot}$				•				SI	,	Gá	AS	InP
Flow rate	of	ga	se	s,	c	m <sup>2</sup>	<b>/</b> m	ılı	١						
Silane															9
Ammon 1 a															40
Nitrogen															40
Substrate	ten	ıpe	ra	tu	re	١,	•0	:							300
Power, W															30
Pressure,	mto	rr	•												500
Time, min.															5
Frequencie	s ,	k۲	łz												30
		MH													3.56

After deposition of the silicon nitride films, the 50.8-mm wafers were broken. Pieces from the same wafer were measured by Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), ellipsometry, and in situ adhesion and friction apparatuses.

For the adhesion and friction experiments the contacting surfaces of the hemispherical monolithic silicon nitride pin specimens were polished first with diamond power 3  $\mu m$  and 1  $\mu m$  in diameter and then with aluminum oxide power 1  $\mu m$  in diameter. All the specimens in this investigation were rinsed with high purity ethanol before the experiments. The radius of curvature of the hemispherical monolithic silicon nitride pin specimens was either 1.6 or 3.2 mm.

Procedures

The adhesion experiments were conducted in vacuum. The pin and flat specimens were placed in a vacuum chamber (Fig. 1), and the system was evacuated and baked out to achieve a pressure of 30 nPa. Further, the hemispherical monolithic silicon nitride pin specimens were ion-sputter etched with a 3000-eV beam at 25-mA current with an argon pressure of 0.7 mPa. The ion beam was continuously rastered over the specimen surface for 20 min.

After sputter etching, the system was reevacuated from 0.7 mPa to a pressure of 30 nPa or lower. The in situ adhesion and friction experiments were conducted with the as-received plasma-deposited silicon nitride films in contact with the ion-sputter-cleaned hemispherical monolithic silicon nitride pin specimens in a 30-nPa vacuum. The surface cleanliness of the pin and flat film specimens was examined by XPS analysis.

For in situ adhesion measurements in vacuum, the flat specimen was brought into contact with the pin specimen by moving the micrometer headscrew forward manually. Contact was maintained for 30 sec; then the pin and flat specimen surfaces were pulled apart by moving the micrometer headscrew backward. An LVDT monitored the displacement of the pin specimens.

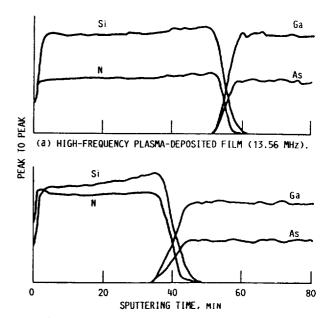
In situ friction experiments were conducted with loads up to 1.7 N that were applied to the pin-flat contact by mechanically deflecting the beam through precision manipulators. Moving the beam in a vertical direction parallel to the flat surface initiated sliding. To obtain consistent experimental conditions, the time in contact before sliding was kept constant at 30 sec. Friction force was continuously monitored during a friction experiment. The sliding velocity was 3 mm/min, and the total sliding distance was about 5 mm. All adhesion and friction experiments were conducted in ultrahigh vacuum.

The ellipsometric measurements were done on a rotating analyzer ellipsometer with a xenon arc lamp. Measurements were made on silicon nitride films grown at 30 kHz or at 13.56 MHz on silicon, gallium arsenide, and indium phosphide substrates. The procedures of the surface chemical analyses are described in detail by Miyoshi et al. (1985), Miyoshi et al. (1987a), Miyoshi et al. (1987b), and Pouch et al. (1987).

## RESULTS AND DISCUSSION

## Silicon Nitride Films

AES analysis provided complete elemental depth profiles for the silicon nitride films deposited by low- and high-frequency plasmas (30 kHz and 13.56 MHz) as a function of the sputtering time. Typical examples are presented in Fig. 2 parts (a) and (b), respectively. Comparison of the low- and high-frequency plasma-deposited silicon nitride films indicated a higher Si/N ratio for the film deposited at 13.56 MHz than for that deposited at 30 kHz.



(b) LOW-FREQUENCY PLASMA-DEPOSITED FILM (30 kHz).
FIG. 2. - AES DEPTH PROFILE FOR PLASMA-DEPOSITED SILICON NITRIDE FILMS ON GALLIUM ARSENIDE.

The silicon nitride films deposited by low- and high-frequency plasmas were also probed by XPS. The Si/N ratios of the argon ion sputter-cleaned silicon nitride films are presented in Table II. Silicon to nitrogen ratios were much greater for the films deposited at 13.56 MHz than for those deposited at 30 kHz and, thus, supported the AES data.

TABLE II. - RATIO OF SI/N IN SILICON

Substrate	Plasma deposition frequency							
	30 kHz	13.56 MHz						
Si	1.1	1.4						
GaAs	1.2	1.4						
InP	1.1	1.7						

Film thickness determined with a rotating analyzer ellipsometer are presented in Table III. The error margins are the 90 percent confidence limits. The results were obtained by using a model which assumed a single film on a substrate. The small error margins showed that this model is an excellent description of the sample.

TABLE III. - FILM THICKNESS OF SILICON NITRIDE FILMS

Substrate	Plasma deposition frequency							
	30 KHz	13.56 MHz						
Si	77.1±0.2	85.6±0.2						
GaAs	77.0±0.1	88.6±0.7						
InP	76.1±0.1	76.6±0.1						

Representative results of the refractive index  $\eta$  and the absorption coefficient  $\alpha$  (where  $\alpha=4\pi K/\lambda$ , K is the extinction coefficient, and  $\lambda$  is the wavelength) for two films on gallium arsenide are presented in Fig. 3. The refractive index for the films made at 13.56 MHz was higher than that of pure amorphous silicon nitride (Palik, 1985); this indicated that a small, but not insignificant, amount of amorphous silicon with its higher refractive index was present. In addition, pure silicon nitride does not absorb at all above 300 nm, whereas amorphous silicon does show absorption in the wavelength range used here. This fact and the results shown in Fig. 3(b), indicating absorption in the film deposited by 13.56 MHz, reinforced our conclusion that amorphous silicon was present in these films.

The films made at 30 kHz have lower refractive indexes than pure silicon nitride and almost vanishing absorption. This indicated the films contained either a small number of voids or a small amount of oxygen, and a negligibly small amount of amorphous silicon. (Oxygen usually exists in the form of silicon oxinitride.)

Results of measurements of silicon nitride films on silicon and indium phosphide are similar to those obtained of silicon nitride on gallium arsenide. Thus, the difference in the properties of high- and low-

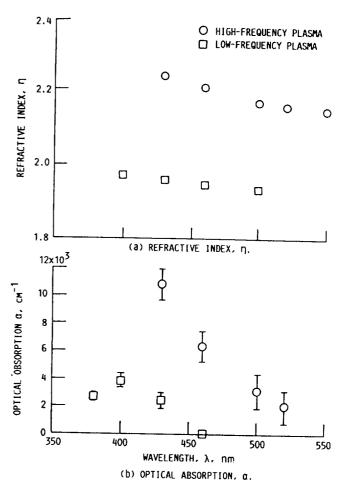


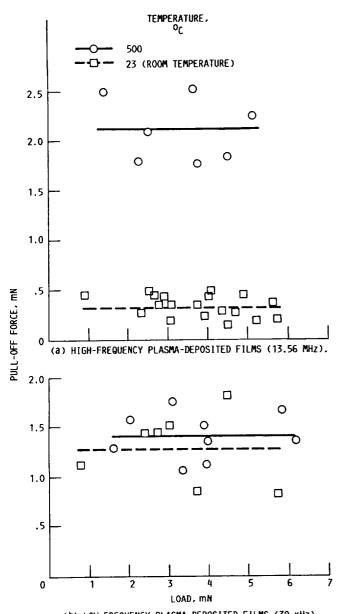
FIG. 3. - REFRACTIVE INDEX AND OPTICAL ABSORPTION OF PLASMA-DEPOSITED SILICON NITRIDE FILMS ON GALLIUM ARSENIDE AS A FUNCTION OF WAVELENGTH.

frequency deposition is due only to the plasma, since there is no indication of substrate interaction.

## Adhesion

Adhesion experiments were conducted with plasmadeposited silicon nitride films in contact with monolithic magnesia-doped silicon nitride at temperatures to 700 °C in ultrahigh vacuum. The strength of adhesion is expressed as the force necessary to pull the surfaces apart; it is called the pull-off force to distinguish it from both thermodynamic adhesion and surface forces (Tabor, 1983). The monolithic silicon nitride pins were sputter-cleaned with argon ions before adhesion experiments. The plasma-deposited silicon nitride films were in the as-received state after they had been baked out in the vacuum system. At room temperature, adsorbates from the environment are present on the surface of the plasma-deposited silicon nitride films.

Figure 4 represents typical plots of the pull-off force (adhesion) as a function of load. The plots indicate no significant change in the pull-off force with respect to load over the load range of 1 to 6 mN. The data, however, indicate clearly that the adhesion depends on the temperature and the plasma deposition frequency.



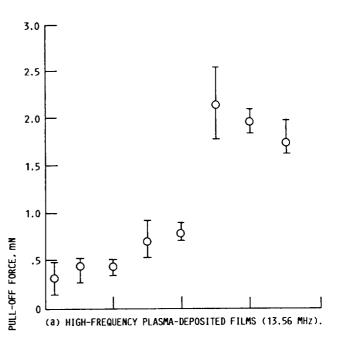
(b) LOW-FREQUENCY PLASMA-DEPOSITED FILMS (30 kHz).

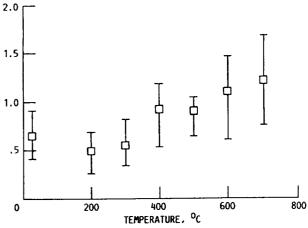
FIG. 4. - PULL-OFF FORCE (ADHESION) AS A FUNCTION OF
LOAD FOR PLASMA-DEPOSITED SILICON NITRIDE FILMS IN
CONTACT WITH HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS IN VACUUM.

Figure 5 presents the average pull-off forces for the as-received silicon nitride films deposited by high- and low-frequency plasmas as a function of temperature. Although the pull-off force (adhesion) for the high-frequency plasma-deposited silicon nitride films increased slightly with temperatures up to 400 °C, it generally remained low at these temperatures. The pull-off force increased significantly at 500 °C and remained high in the range of 500 to 700 °C. Very strong adhesive bonding can take place at the contacting interface at temperatures in the range of 500 to 700 °C.

On the other hand, the adsorbed carbon contaminant (determined by XPS) decreased rapidly with increasing temperature to 400 °C. Above 400 °C it had disappeared from the surface of the silicon nitride film. The rapid increase in adhesion at 500 to 700 °C can be related to the removal of the adsorbates, resulting in a strong surface chemical reaction between the monolithic silicon nitride and the plasma-deposited silicon nitride film; whereas the low friction below 400 °C was associated with the presence of adsorbates.

For the silicon nitride films deposited by low-frequency plasma (30 kHz), the pull-off force increased with increasing temperature (Fig. 5(b)). When compared with the results of Fig. 5(a), however, the pull-off forces were generally lower in the high temperature range of 500 to 700 °C.

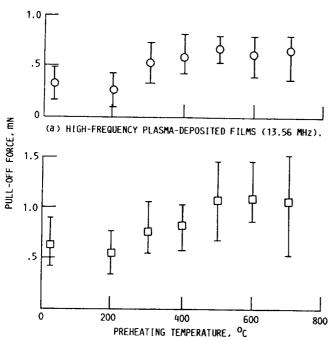




(b) LOW-FREQUENCY PLASMA-DEPOSITED FILMS (30 kHz).

FIG. 5. - PULL-OFF FORCE (ADHESION) AS A FUNCTION OF TEMPERATURE FOR PLASMA-DEPOSITED SILICON NITRIDE FILMS IN CONTACT WITH HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS IN VACUUM.

Figure 6 presents the average pull-off forces for the silicon nitride films measured at room temperature in ultrahigh vacuum. The as-received films (after bake out) were preheated at various temperatures in a 30-nPa vacuum. The temperatures given in Fig. 6 were the highest temperatures to which the silicon nitride films had been preheated. The adhesion experiments represented in Fig. 6 were conducted at room temperature in 30-nPa vacuum. Figure 6 shows the effect of adsorbates on the surface characteristics of the silicon nitride film. The pull-off force increased with increasing preheating temperature. The pull-off force for preheating temperatures above 500 °C was almost twice that for the low preheating temperatures. The high adhesion for specimens preheated at temperatures above 500 °C correlated with the removal of adsorbates from the surfaces of silicon nitride films and thus supported the XPS data.



(b) LOW-FREQUENCY PLASMA-DEPOSITED FILMS (30 kHz).

FIG. 6. - PULL-OFF FORCE (ADHESION) FOR PLASMA-DEPOSITED SILICON NITRIDE FILMS IN CONTACT WITH HEMISPHERICAL MONOLITHIC SILICON NITRIDE PIN IN VACUUM AT ROOM TEMPERATURE. FILMS WERE PREHEATED AT VARIOUS TEMPERATURES TO 700 OC.

## Friction and Wear

Friction force traces resulting from sliding were characterized by both stick-slip and randomly fluctuating behavior, regardless of plasma deposition frequency.

High-frequency plasma-deposited silicon nitride. Typical plots of the coefficient of friction for the high-frequency plasma-deposited silicon nitride films as a function of load are presented in Fig. 7. There are actually two friction modes involved in the tribological process with regard to normal load, plasma deposition frequency, and sliding temperature. The first mode is that in which the coefficient of friction is not constant, but decreases as the load increases (Fig. 7(a)). The sliding action of the silicon nitride pin on the silicon nitride film produces predominantly

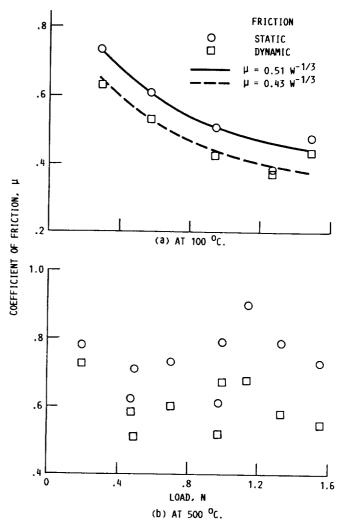


FIG. 7. - COEFFICIENT OF FRICTION AS A FUNCTION OF LOAD FOR HIGH-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILMS IN SLIDING CONTACT WITH A HEMISPHERICAL MONOLITHIC SILICON NITRIDE PIN IN VACUUM AT TEMPERATURES OF 100 AND 500 OC.

elastic deformation. The real area of contact, therefore, is not generally proportional to the load, and there is a corresponding deviation from Amontons' Law. A first approximation for the load range investigated gives the relation between coefficient of friction  $\mu$  and load W as  $\mu = kw^{-1/3}$  (Bowden and Tabor, 1950; Bowden and Tabor, 1964; and Miyoshi and Buckley, 1979). The inverse negative three power may be interpreted, most simply, as arising from an adhesion mechanism; and the area of contact is determined by elastic deformation. Friction is a function of the shear strength of the elastic contact area.

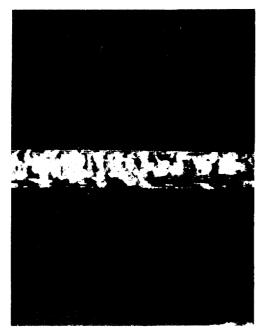
The second mode characterizes friction force by fluctuating behavior with respect to the normal load, as typically presented in Fig. 7(b).

High-frequency plasma-deposited silicon nitride films behaved in the first friction mode, as typically presented in Fig. 7(a), at sliding temperatures to  $400\,^{\circ}\text{C}$ .

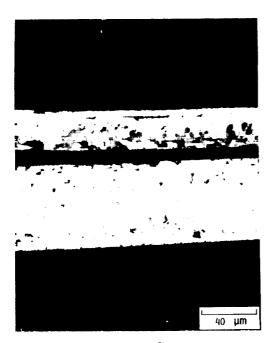
When the temperature was further increased to the range of 500 to 700 °C, the coefficient of friction was characterized by the second mode, that is, the fluctuating behavior with normal load (Fig. 7(b)). Under such

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conditions, the sliding action produced failure in the film and at the interfacial adhesive bonds between the film and substrate and caused breakthrough of the film in the contact area (Fig. 8). Also, wear debris particles of silicon nitride film were observed. Thus, the increase in friction at higher loads was caused by the



(a) AT 500 °C.



(b) AT 700 OC.

FIG. 8. - OPTICAL PHOTOMICROGRAPHS OF WEAR TRACKS GENERATED BY HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS AT HIGH TEMPERATURES IN VACUUM ON HIGH-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILM SURFACES.

gross failure of the silicon nitride film. A large amount of the friction energy was dissipated in the failure process of the film during sliding.

The coefficient of friction for the silicon nitride films deposited by high-frequency plasma as a function of sliding temperature is indicated in Fig. 9. The static friction characteristics (Fig. 9(b)) are the same as those of adhesion presented in Fig. 5(a). The coefficient of static friction increased slightly with increasing temperatures to 400 °C and rapidly with increasing temperatures above 500 °C, remaining high in the range of 500 to 700 °C. The trend for the coefficient of dynamic friction is also quite similar to that of adhesion. When compared with the static friction, the coefficient of dynamic friction was generally lower at temperatures to 700 °C.

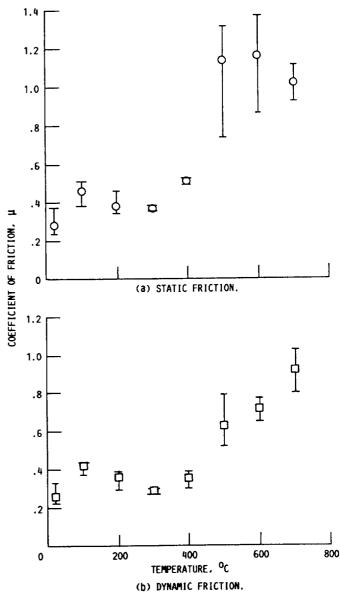


FIG. 9. - EFFECT OF TEMPERATURE ON COEFFICIENT OF FRICTION FOR HIGH-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILMS IN SLIDING CONTACT WITH HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS IN VACUUM.

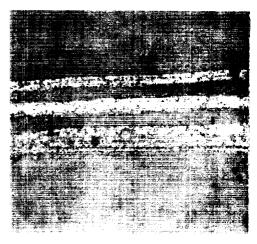
Low-frequency plasma-deposited silicon nitride films. The friction behavior of the low-frequency plasma-deposited silicon nitride film was characterized by the first mode, as indicated in Fig. 10, regardless of sliding temperature and failure of the film. Optical microscopic examination of the wear tracks on the silicon nitride films clearly revealed that the sliding action produced failure in the film and at the interface between the film and substrate and caused breakthrough of the film in the contact area at temperatures of 500 and 700 °C (Fig. 11). However, the films at 500 and 700 °C provided somewhat lower coefficients of friction than were obtained for high-frequency plasma-deposited films, thus the low-frequency plasma-deposited films had less resistance to shear (cohesively weaker) and less adherence to the silicon substrate.

The coefficient of friction for the silicon nitride films deposited by low-frequency plasma as a function of sliding temperature is indicated in Fig. 12. The static friction characteristics (Fig. 12(a)) are similar to

FRICTION .6 0 STATIC DYNAMIC .5  $= 0.3 \text{ W}^{-1/3}$  $= 0.44 \text{ W}^{-1/3}$ .3 8 8 .2 COEFFICIENT OF FRICTION, (a) AT ROOM TEMPERATURE, 23 Oc. ·8 📵 .7 .6 .5 . 4 .3 1.0 LOAD, N (b) AT 500 OC.

FIG. 10. - COEFFICIENT OF FRICTION AS A FUNCTION OF LOAD FOR LOW-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILMS IN SLIDING CONTACT WITH HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS IN VACUUM AT ROOM TEMPERATURE AND 500 Oc.

those of adhesion presented in Fig. 5(a). The coefficient of static friction increased slightly with increasing temperatures to 500 °C and quite rapidly at 500 °C; it remained high at 600 and 700 °C. The coefficient of dynamic friction was also similar to that of adhesion. When compared with the static friction, the coefficient of dynamic friction was generally lower for temperatures up to 700 °C.



(a) AT 500 °C.







(b) AT 700 OC.

FIG. 11. - OPTICAL PHOTOMICROGRAPHS OF WEAR TRACKS GENERATED BY HEMISPHERICAL MONOLITHIC SILICON NITRIDE PINS AT HIGH TEMPERATURES IN VACUUM ON LOW-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILM SURFACES.

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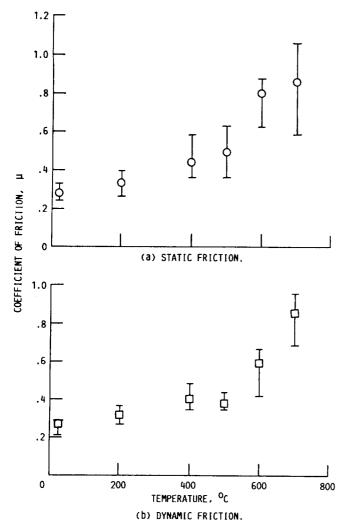


FIG. 12. - EFFECT OF TEMPERATURE ON COEFFICIENT OF FRICTION FOR LOW-FREQUENCY PLASMA-DEPOSITED SILICON NITRIDE FILMS IN SLIDING CONTACT WITH HEMISPHERICAL MONOLITHIC PINS IN VACUUM.

### CONCLUSIONS

Adhesion and sliding friction experiments were conducted with silicon nitride films (deposited by low- and high-frequency plasma, 30 kHz and 13.56 MHz) in contact with a hemispherical monolithic magnesia-doped silicon nitride pin at temperatures to 700 °C in vacuum. As a result of these experiments the following conclusions are drawn:

- 1. There was a significant temperature influence on adhesion, friction, and wear of the silicon nitride films.
- 2. The Si/N ratios for the silicon nitride films deposited at 13.56 MHz were much greater than for those deposited at 30 kHz. Amorphous silicon was present in both low- and high-frequency plasma-deposited silicon nitride films. There was, however, more amorphous silicon in the films deposited at 13.56 MHz than in those deposited at 30 kHz.

- 3. The adhesion behavior of the high-frequency plasma-deposited silicon nitride films at temperatures to 700 °C was similar to that of the coefficient of friction. Although the adhesion and friction remained low at temperatures to 400 °C and the effect of temperature was small, they increased greatly at temperatures above 500 °C.
- 4. The adhesion behavior of the low-frequency plasma-deposited silicon nitride at temperatures to 700 °C was similar to that for the coefficient of friction. The adhesion and friction remained low at temperatures to 500 °C and increased for temperatures above 600 °C.
- 5. The silicon nitride film wear occurred in the contact area at high temperatures. The wear correlated with the increase in adhesion and friction for the lowand high-frequency plasma-deposited films above 600 and 500 °C, respectively.
- 6. The low- and high-frequency plasma-deposited thin silicon nitride films exhibited a capability for lubrication (low adhesion and friction) in vacuum at temperatures to 500 and 400 °C, respectively.

## **ACKNOWLEDGEMENT**

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the investigation indicated those deposited at 30 kHz. silicon nitride films. However, those deposited at 30 kHz. films. Wear occurred in the and friction for the low- an	kHz and hat the Si/li Amorphou yer, more a Temperatu contact and high-frequency	13.56 MHz) at variou N ratios were much a silicon was present amorphous silicon occure significantly influere at high temperature plasma-deposited thin silicon nitrical.	is temperatures greater for the in both low- a curred in the finced adhesion, re. The wear could films above the films exhibit the state of the stat	licon nitride films deposited by low- and to 700 °C in vacuum. The results of films deposited at 13.56 MHz than for and high-frequency plasma-deposited lms deposited at 13.56 MHz than in friction, and wear of the silicon nitride correlated with the increase in adhesion to 600 and 500 °C, respectively. The sed a capability for lubrication (low sectively.
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